APPLICATION FOR UNITED STATES LETTERS PATENT

for

COMPOSITION AND METHOD FOR A DUAL-FUNCTION SOIL-GROUTING EXCAVATING OR BORING FLUID

by

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This application is a continuation-in-part of U.S. Patent Application No. 09/023,150, filed February 12, 1998 which in turn claims priority of U.S. Provisional Application No. 60/037,712 filed February 12, 1997.

BACKGROUND OF THE INVENTION

In earth boring and excavating for wells, deep foundations, tunnels and other geotechnical applications, fluids or muds have been used to hold open and maintain the stability of boreholes and excavations. These fluids or muds have used hydrostatic pressure and controlled interaction with the earth to accomplish their functions. The excavations have been kept full of the fluids or muds during the excavating or boring process, with or without circulation of the fluids.

Separately, in processes for improving the cohesion and load-bearing properties of granular or unconsolidated soils and other unstable granular earth formations or materials, reactive compositions have been injected into and mixed with the soils to cause solidification or hardening of the soils. These reactive compositions have comprised silicates cementitious grouts and other materials. The application of these soil-improvement materials and techniques has been done as a prelude to excavating, drilling, tunneling, or pile-driving, in order to render the soils resistant enough to support deep excavations for things such as foundation systems such as bored piles, or to bear the weight of structures erected on pad-type foundations or spread footings. These processes whereby weak soils are prepared to receive excavations for things such as foundation systems or other geoconstruction elements are generally referred to as ground improvement.

In a typical sequence of events for the construction of structures on poor soil, ground improvement techniques are used, followed by excavating or drilling to create deep foundation elements such as diaphragm wall panels, barrettes, or bored piles. Frequently the excavations or borings are made with the help of a fluid or mud as described above. In this two-step process the weak soil is first strengthened by ground improvement techniques such as reactive silicate injection or mixing, then excavations are created in or through the strengthened soil with the help of an excavating fluid or drilling mud. Finally, reinforced concrete is formed in the excavations in order to create a competent deep construction system.

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In the prior art, silicates and silicate-reactive compounds have been injected into or mixed with granular, rubbleized or vugular earth formations, fills or other materials in advance of or during pauses in drilling or excavating, to strengthen or solidify the earth formations. Polymer-based fluids have been used for excavating and drilling, to support the walls of the excavations or wells. And silicates have been added to drilling muds in attempts to prevent heaving of shales. What is unknown in the prior art is the formulation and effective application of a single fluid which is both and at the same time a drilling mud or earth support fluid and a reactive, soil-permeating, silicate-based chemical-grouting ground-improvement or ground-solidification agent which is effective in the presence of unstable earth environments (e.g. sand).

As a result of the above, there remains and exists an unmet need for fluids that stabilize and/or make possible the excavation in the earth. The instant invention offers an improvement over the prior arts for both the stabilization of boreholes, earth excavations and the like; and ground improvement. An adaptation of the invention is useful during the preparation of guide-walls for diaphragm walls and the solidification of near-surface zones of loose soil.

SUMMARY OF THE INVENTION

This invention relates to fluids for use in boring and excavating operations. More specifically, this invention relates to earth-stabilization and earth-support fluids, their composition, and techniques for preparing, using, and maintaining them. The compositions and methods are useful in creating boreholes, tunnels and other excavations in unstable soils and earth formations, especially those composed partially or wholly of sand, gravel or other granular or permeable material. The fluids of the invention, when used according to the methods of the invention, have unique dual functionality as excavating fluids and as earth-grouting or soil-hardening compositions.

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These and other features of the present invention are more fully set forth in the following description of preferred or illustrative embodiments of the invention.

DESCRIPTION OF ILLUSTRATIVE EMBODIMENTS

The instant invention is a composition and method of application for a dual-purpose excavating and soil-strengthening fluid composed of: water; water-dispersible polymers; alkalis; optionally soil or earth solids; various forms of sodium silicate; and, optionally, sodium aluminate, calcium chloride, carbon dioxide and chlorine gas, citric, sulfamic or other acids and salts thereof salts, or other crosslinking or catalytic agent which assists in making the sodium silicate - slurry solution somewhat insoluble to totally insoluble. The fluid's multi-purpose nature is expressed in its functions as (1) an earth-support fluid as known in the prior art, and (2) a soil-strengthening fluid which functions in a manner similar to silicate "chemical grouts" known in the prior art (3) a weighting agent to increase the specific gravity of a slurry system. The novelty lies in the accomplishment of the earth support function (as performed by drilling muds, etc.) concurrently and in combination with the chemical grouting or ground improvement function (as previously performed by reactive silicate injection and/or soil mixing prior to excavating or boring).

The fluids of the invention are preferably based on aqueous dispersions of water-dispersible polymers, and may contain inorganic buffers, polycationic additives, soil or mineral solids and other materials as disclosed in the prior art and in U.S. patents numbers 5,407,909 and United States Patent 5,663,123 the contents of which are hereby incorporated herein by reference. In expressing the current invention, these prior-art fluids are modified by dosing with sodium silicate and, optionally, sodium aluminate. The silicate, by being present in the excavating fluid, permeates the weak or unstable layers of granular earth material or fills which are penetrated by the excavating or boring machinery. The silicate reacts with the naturally occurring soil components under excavation, along with any introduced crosslinking or catalytic agents. The degree of strengthening, increased cohesion, or hardening is_developed by enhancing or preventing alteration of weak bonds among the granular earth material, or by forming a glasslike siliceous matrix within the soils present. This effect is achievable in granular formations and soils such as gravel and sand; in filled areas and irregular materials such as rubbleized concrete and mixed fills in and around

old foundation systems; in sand-bearing soils such as clayey sand, sandy clay, silty sand and sandy silt; and in other permeable, clastic, granular or partially-granular earth formations such as glacial tills, oolite, shell beds, vugular or fractured rocks, rock washes and decomposed rock materials.

Because the fluids of the invention are low-solids fluids based on polymers, the whole fluid, when formulated with lower concentrations of or without fluid loss control provisions or additives, is able to permeate sandy earth formations more freely than can bentonite-based fluids, which deposit a low-permeability cake on the face of the formation. The bentonite filter cake allows principally water to pass into the formation, leaving most of the colloidal or water thickening constituents of the fluid in the filter cake. The soil-permeation characteristics of polymer-based fluids facilitate the fluids' ground improvement functionality when silicates are present in the fluids, because the silicates (and optional aluminates) are carried by the permeating whole fluid into the pore system of the formation surrounding the excavation. The fluids of the invention can also incorporate bentonite at up to about 3% wt./vol. Such polymer-extended, bentonite-containing fluids can exhibit soil-permeation characteristics sufficiently similar to pure polymer fluids that they can be useful in expressing the excavating fluid or composition of the invention, as well as the methods of the invention.

Fluids containing bentonite at concentrations greater than about 3% can also be used to express a method, if not a composition, of the invention. When bentonite-based fluids or other fluids based on finely-divided solids are dosed with the silicates, hydroxides and optional aluminates of the invention and used to drill or excavate the weak, loose or unconsolidated permeable earth formations discussed herein, the silicate-bearing filtrate from these fluids can permeate the formations and strengthen them by the mechanisms discussed.

The time required for the silicate to react with and significantly increase the stability of the earth formation is sufficiently short as to be useful to the excavator or driller to improve the efficiency of the excavating process or allow for the continuation of excavation in the absence of traditional soil stabilization pretreatments such as grouting or post-treatments such as backfilling with earth, lean

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mix, or concrete. This improvement not only significantly impacts the logistics of excavating unstable soil, but reduces the overall cost of the excavation process.

This differs from classical methods of soil stabilization (ground improvement) wherein silicate compounds and usually calcium bearing agents and other compounds were separately injected into the ground with specially-designed equipment to stabilize the earth formation prior to attempting excavation or other steps in the geoconstruction process. This process of ground improvement is currently practiced prior to beginning many types of boring, excavating or geoconstruction. Until now it was always assumed that the soil needed to be stabilized prior to excavation, to make it excavable. In the excavation and construction of structures such as tunnels, barrettes, bored piles and slurry walls, the prior step of ground improvement may now be eliminated in many cases through the use of the present invention. The invention allows the direct excavation and simultaneous strengthening of unstable, low cohesion or weak zones or areas. The invention is thus useful and cost-beneficial to the industry.

This invention provides a novel method of delivery of a ground-improvement system in a practical and especially efficient manner that incorporates ground improvement into the process of excavating or boring. The invention adds strength to a freshly-excavated area that will last long enough to keep its shape through the completion of concrete placement (or the placing of casing or other downhole components, in the case of wells). It is compatible with polymer fluid systems currently in use in the industry, as well as with fluids based on bentonite and other finely-divided solids. One of the main uses of the invention is in bringing about adequate stability to running sands and loose earth layers typically containing mineral materials in an unstable mixture that is capable of sloughing or collapsing into the freshly cut or drilled areas.

This invention comprises the addition of about 0.1% to about 50.0% by weight of sodium silicate to the drilling/excavating fluid, using any of the commercially available forms of the chemical. The higher ratios of silicon dioxide to sodium oxide are preferred due to economy and utility.

The silicate most commonly employed in the treatment of building materials is a solution with a silica: alkali ratio of about 3.22 and is sold at a density of about 41

degrees B at about 68°F or a specific gravity of about 1.39 to about 1.38. Such a product is known to be Grade N ® from Philadelphia Quartz Company or Grade 40 ® from Oxychem. Inc. of New York. Illustrative commercially available silicate solutions and their composition are given below in Table 1.

TABLE 1.

		3)	COOL COOL COOL	(1)00	Viscosity
		Wt. Katio	,	(3	Viscosity
Producer	Product Name	SiO_2/Na_2O	$\%Na_2O$	$\%SiO_2$	ر آ	- 1	g/cm	Centipoises
PO	STIXSO® RR	3.25	9.2	30.0	42.7	11.8	1.41	830
PO	N®	3.22	6.8	28.7	41.0	11.6	1.38	180
PO	E®	3.22	8.6	27.7	40.0	11.5	1.37	100
PO PO	®O	3.22	9.1	29.5	42.2	11.8	1.41	400
PO	K®	2.88	11.0	31.7	47.0	12.3	1.47	096
PO	M®	2.58	12.4	32.1	49.3	12.6	1.50	780
PO	STAR®	2.50	10.6	26.5	42.0	11.7	1.40	09
PO PO	RU®	2.40	13.8	33.2	52.0	13.0	1.55	2,100
PO	$\mathcal{D}^{@}$	2.00	14.7	29.4	50.5	12.8	1.53	400
PO	C®	2.00	18.0	36.0	59.3	14.1	1.68	70,000
PO	STARSO®	1.80	13.4	24.1	44.6	12.0	1.43	09
OXY	40	3.22	9.1	29.2	41.5	11.67	1	206
OXY	40 Clear	3.22	9.1	29.2	41.5	11.67	1	206
OXY	42	3.22	9.3	30.0	42.5	11.78	1	385
OXY	JW-25	2.54	10.6	26.9	42.0	11.73	1	63
OXY	JW Clear	2.54	10.6	26.9	42.0	11.73	1	63
OXY	47	2.84	11.2	31.9	47.0	12.33	1	889
OXY	49 FG	2.58	12.4	32.1	49.0	12.58	ı	633
VXO	50	2.00	14.7	29.4	50.0	12.71	1	336
VXO	52	2.40	13.9	33.4	52.0	12.98	i i	1760
VXO	WD-43	1.80	13.1	23.6	43.8	11.84	1	69
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Information provided by PQ Corporation Typical Properties of Sodium Silicates Information and Occidental Chemical

Corporation Literature

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There are four principal means of employing the sodium silicate in the practice of the invention. These, for purposes of explanation, are herein referred to as: (1) "Dilute Neat"; (2) "Concentrated Neat", (3) "Concentrated Accelerated", and (4) "Dilute Accelerated".

"Dilute Neat" denotes the addition of sodium silicate in relatively small concentrations (about 0.1% to about 10% by weight) to part or all of the volume of the fluid in use. The silicate acts as an additive to the system, and the silicate-treated excavating or boring fluid permeates the earth formation adjacent to the excavation in proportion to the conductivity of the formation to the fluid at the extant hydrostatic pressure differential. Silicate addition enhances the excavating or boring fluid's characteristics by developing a concentration gradient that increases the rate of fluid mobility into the surface walls of the excavation. The fluid can be any of the polymerbased or polymer-extended fluids known to those skilled in the art, with the added requirement that the fluid pH be maintained in the alkaline range of about 9 to about 13. The natural acidity or buffering capacity of the soil ideally causes a drop in pH of the permeating fluid, causing precipitation of silica onto the mineral surfaces and into the pore spaces of the formation adjacent to the borehole or excavation. Even in the absence of a sufficient drop in pH to precipitate silica, the silicate-bearing fluid can prevent destabilization of sandy formations. This is accomplished by a presumed action of preserving or reinforcing the weak hydrogen or silica-hydroxyl bonds between the grains of the formation.

The "Concentrated Neat" formulation and method consists of establishing a higher concentration (greater than about 10% by weight) of silicate in part or all of the volume of the fluid in use. This is typically accomplished by adding such a concentration of sodium silicate directly to that portion of the fluid which is, at the time of interest, in contact with the zone requiring stabilization. One method is to pour or deliver concentrated sodium silicate into the fluid in the open mouth of the excavation, and wait to allow time for the heavy silicate to sink to the lower portion of the excavation, thus establishing a "site-specific" concentration gradient that delivers a suitably high dose of silicate in that portion of the fluid which is involved in cutting into the unstable

formation. Other methods of selectively delivering high doses ("slugs") of silicate to portions of the fluid column in a borehole or excavation are known to those skilled in the arts of excavating and drilling. (For example, in rotary-type well-drilling in which a circulating fluid is used, the conventional technique for "spotting" a liquid opposite a zone of interest may be used.) A Concentrated Neat application can also involve adding a suitable concentration of silicate to the entire volume of fluid in use, or boosting the silicate content of only a portion of a Dilute Neat system.

A "Concentrated Accelerated" application comprises the use of more than 10% by weight of silicate in all or a portion of the fluid in use, and further adding sodium aluminate or other analogous aluminate at up to and in excess of about 10% by weight to cause enhanced solidification of the soil/fluid mixture or of the fluid-permeated earth material. Such a mixture of suitable earth or mineral materials (such as sand, sandy soil, gravel, decomposed rock, granular mineral fills, rubbleized or pulverized concrete, etc.) with water, alkali, polymers, sodium silicate and optionally sodium aluminate, will solidify to a stable mass in from a few minutes to several hours time. This Concentrated Accelerated system will require the presence of sodium aluminate from about 0.1% to greater than about 10.0% by weight of the fluid solids. The use of the Concentrated Accelerated system can allow excavation to continue almost without delay. Setting times for the Concentrated Accelerated system can be as little as about 30 seconds after the delivery and incorporation of the sodium aluminate into the portion of the silicate-treated fluid that is in contact with the zone of interest.

The fourth expression of the system, known herein as "Dilute Accelerated", comprises a fluid with a silicate content of less than about 10% by weight, dosed additionally with sodium or analogous aluminate at up to about 10% by weight. This dilute system is not expected to be as effective as the Concentrated Accelerated system due to a deficiency of silicate with which the aluminate can react. But it nonetheless may provide some useful functionality or benefit over the Dilute Neat system in some situations.

The instant invention can be summarized as: a composition and method of delivery of a soil-repairing or earth-strengthening excavating or boring fluid to a zone or

layer where it is needed during the process of excavating or boring, to strengthen, harden or partially harden an incompetent, sloughing or running zone or layer of sand, sandy soil, or other granular mineral-bearing material in the earth. In order to differentiate the instant invention from previous efforts in the well-drilling art to use silicate-treated drilling fluids to stabilize shales, it can be specified that the instant invention is useful in earth formations or other zones of mineral-bearing materials found in the earth which are significantly permeable (permeability greater than about 100 millidarcies) and of relatively low compaction. That is to say, a significant portion by weight of the subject earth materials or formations has a grain size larger than about 20 micrometers (silt), and probably also a significant portion has a grain size larger than about 70 micrometers (sand), and compaction less than about 50 blows per foot, standard penetration, NPT. The invention is also expected to be useful in stabilizing weak or poorly-cemented sandy formations when a significant portion by weight of the subject earth materials or formations has a grain size larger than about 70 micrometers, with compaction higher than about 50 blows per foot, as might occur in well-drilling operation.

At the conclusion of concrete placement the final returned fluid of this invention from an excavation and also during the course of drilling a loaded soil or fluids no longer needed and excess fluids may be utilized around a project site in the useful manner of spray application to control dust in dry seasons or in areas of high concern about dusts from the project site.

Additionally the polymer based fluids of this invention may be used to control erosion by distributing around the site to soak into surface soils to aid in at least a partial consolidation to minimize loose soil run off.

The method of delivery of an excavation composition or soil repair fluid to a zone of an excavation or borehole wherein the excavation composition or soil repair fluid contains silicate salts at concentrations of about 0.1% to about 30% by weight of the total excavation composition, with alkaline hydroxides from about 0.01% to about 10.0% and aluminate salts from about 0.0% to about 30.0% of the fluid. The balance of the fluid is preferably water and small amounts of various water-dispersible polymers known to the industry (e.g. polyacrylamides, cellulosics, guars, starches, xanthan, et al), in addition

finely-divided solids from the soils being excavated. The fluid may also be based on bentonite, other clays or finely-divided solids (e.g. hematite, barite, mixed clay, kaolin, calcium carbonate), or a combination of bentonite and polymers or other finely-divided solids and polymers.

Such a composition, when used in the excavation, drilling or treatment by permeation of incompetent, sloughing or running zones or layers of sand, sandy soil, sea shells, gravel, decomposed rock or concrete, or other granular mineral-bearing material in the earth, will constitute the delivery of a soil stabilization system for the purposes of this invention to enable the strengthening of the earth formation or material immediately surrounding or adjacent to the excavation or borehole. This fluid system allows stabilization or the solidification of incompetent or sloughing granular earth or mineral material to enable further drilling or excavating followed by such processes as in-situ formation of concrete foundation elements or the completion of wells in previously unconsolidated sands and soils. The silicates, hydroxides and aluminates are preferred to be in the sodium forms, but potassium, ammonium, and others such as magnesium, iron and calcium are useful.

One advantage of this instant invention is the ability to stabilize extremely weak zones without the use of cementitious mixtures based on Portland cement or other calcium- or magnesium-bearing compounds (e.g. gypsum cement), which may take longer to set and which can contaminate drilling or excavating fluids with calcium or other divalent cations during subsequent re-excavation or drilling operations. Calcium contamination can damage most types of drilling or excavating fluids, resulting in increased cost and time expenditure to remedy the effects of such contamination. The system described in the instant invention can be formulated entirely without calcium or other divalent cations.

Given the above, one illustrative embodiment of the present invention is an excavation fluid composition useful for enlarging a cavity in the earth. The excavation fluid includes a synthetic polymer and sodium silicate. The composition is formulated so as to enable the fluid in contact with unstable or sandy soils in the selected areas of the

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excavation to react and form silicate-based derivatives with lesser solubility and movement. This formulation thereby improves soil stability at the excavation wall and enables the excavation of the earthen cavity to continue without the dangers of sloughing or caving in. In an alternative illustrative embodiment of this excavation fluid an aluminate salt is included which increases the speed of reaction of excavation fluid composition with the unstable and sandy soils. In yet another illustrative embodiment of this excavation fluid the composition further includes an alkalinity source. Exemplary alkalinity sources include the sodium and potassium salts of hydroxide and combinations thereof. Preferably the alkalinity source is present from about 0.01% to about 10.0% by weight of the excavation fluid. In yet another illustrative embodiment, the excavation fluid composition the synthetic polymer comprises one or more monomers selected from: acrylamide, methacrylamide, acrylic acid, methacrylic acid, maleic acid, fumaric acid; maleic anhydride, methacrylic anhydride, itaconic acid, acrylic acid dimer(BCEA), Misopropenylbenzyl dimethyl isocyanate and the nonionic associative monomer derivatives, esters or urethane, so produced containing nonionic surfactant starting materials prepared from ethylene oxide and/or, propylene oxide and/or, butylene oxide and/or C_1 to C_{20} alkyl alcohols and/or C_8 to C_{12} alkyl phenols; itaconic acid, vinylsulfonic 2-acrylamido-2-methylpropane sulfonic acid, sulfonic acid, acid, styrene methallylsulfonic acid, vinyl acetic acid, 4-methylpentenoic acid, allylacetic acid, Bhydroxyethylacrylate, x-haloacrylic acid; M- isopropenylbenzyl dimethyl isocyanate and its nonionic derivatives prepared from alkyl alcohols; methylenebisacrylamide, Nmethylol acrylamide, triallyl cyanurate, vinyl crotonate, divinylbenzene, allyl methacrylate; acrylic acid esters of sucrose, hexallyl sucrose, trimethylolpropane triacrylate, ethylene glycol diacrylate, diethylene glycol diacrylate, ethylene glycol dimethacrylate, and the like; methacrylic anhydride esters or maleic anhydride esters of sucrose, sorbitol, sorbitol esters with fatty acids; guar gum, starch, ethylated starch, oxidized starch, starch fatty acid esters, dodecylsuccinic anhydride modified starch, agar gum, xanthan gum, arabic gum or galacto-mannin derivatives prepared from methacrylic anhydride or maleic anhydride or M-isopropenylbenzyl dimethyl isocyanate resulting in hybrid monomers; vinyl acetate, N-vinyl formamide, N-vinyl acetamide,, N-vinyl

pyrolidone, styrene, butadiene, isoprene, chloro-butadiene, vinyl chloride, vinylidene chloride, C₁ to C₂₀ acrylate and methacrylate esters; methacryloxyethyl dimethylamine, methacrylamido propyl dimethylamine, dimethyl diallyl ammonium chloride, diethyl diallyl ammonium chloride, and their methyl sulfate and methyl chloride derivatives and water soluble or dispersible salts and combinations thereof. In a preferred illustrative embodiment the excavation fluid includes a synthetic polymer, sodium silicate that is about 0.1% to about 50.0% of the fluid composition, sodium hydroxide that is about 0.01% to about 10.0% of the fluid composition and sodium aluminate that is about 0.0% to about 30.0% of the fluid composition.

Yet another illustrative embodiment of the compositions of the present invention includes an anhydrous acid solidification mixture including: a structure material used to provide stability, strength, support, foundation, or volume to the solidification mixture and being selected from: sands, soils, clays, pebbles, cobbles, marble, granite, stones, gravel, rocks, bentonite, cement, polymer fibers, sandstone and combinations thereof; a polymer component; an accelerator compound selected from chemicals capable of producing carbon dioxide in acidic environments, chemicals capable of producing chlorine gas in acidic conditions, sodium aluminate, inorganic chloride salts, inorganic sulfate and inorganic sulfite salts; an acidic component selected from solid chemicals that gives H+ in aqueous solutions; and a silicate component selected from sodium orthosilicate, sesquisilicate, metasilicate, disilicate and combinations thereof. In one preferred illustrative embodiment the accelerator compound is selected from the group consisting of potassium and sodium salts of hydrogen carbonate, potassium and sodium salts of carbonate, sodium and potassium hypochlorite, sodium aluminate, and combinations thereof, and wherein said acidic components is selected from the group consisting of citric acid, the salts of citric acid, sulamic acid, and combinations thereof.

A further illustrative composition contemplated herein is an anhydrous alkali solidification mixture. Like the previous acid solidification mixture, the alkali solidification mixture includes a structure material used to provide stability, strength, support, foundation, or volume to the solidification mixture and being selected from:

sands, soils, clays, pebbles, cobbles, marble, granite, stones, gravel, rocks, bentonite, cement, polymer fibers, sandstone and combinations thereof. The composition further includes a polymer component; an accelerator compound, said accelerator compound being selected from sodium aluminate, all inorganic chloride salts, all inorganic sulfate or sulfite salts; a basic component, said basic component being selected from potassium or sodium hydroxide, the sodium or potassium salts of hydrogen carbonate, and carbonate, or any basic salt or solid chemicals that give OH in aqueous solutions; and a silicate component said silicate component being selected from sodium orthosilicate, sesquisilicate, metasilicate, disilicate and combinations thereof.

Both the acid and the alkali solidification mixture described above may be used to stabilize an area under excavation by auger, drill, bucket, clam-shell, continuous cutter, grab and the like that is in the process of preparing a cavity by adding the solidification mixture into a bentonite or polymer based excavation composition with a silicate source present during the process of excavation.

The present invention also contemplates and encompasses the methods of using the fluids described herein in the stabilization of earth excavations. Thus, the present invention is contemplated to include a method of stabilizing the wall of an earthen excavation, the method including placing in said earthen excavation a digging fluid comprising a polymer and sodium silicate. the digging fluid composition is formulated so as to enable the fluid in contact with unstable or sandy soils in the selected areas of the excavation to react and form silicate-based derivatives with lesser solubility, and movement and thus improve soil stability at the excavation wall.

The contemplated methods of the present invention also include an improved method of stabilizing an earth excavation the earth excavation having within it a digging fluid, the digging fluid including water and a polymer. The improvement includes adding a soluble silicate ion source, a soluble hydroxide ion source, and optionally a soluble aluminate ion source to the digging fluid in concentrations sufficient to stabilize said earth excavation. In one preferred embodiment the soluble silicate ion source is selected from the group consisting of the sodium orthosilicate, sesquisilicate, metasilicate,

disilicate and combinations thereof. In another embodiment the soluble hydroxide ion source is selected from the group consisting of the sodium and potassium salts of hydroxide and combinations thereof. In another embodiment the soluble aluminate ion source is selected from the group consisting of sodium, ammonium, potassium, salts of aluminate and combinations thereof. In another embodiment the source of soluble silicate ion is added prior to the addition of the source of soluble aluminate ion.

The methods of the present invention also encompass methods of using the above disclosed compositions to control erosion and dust by aiding in the consolidation of surface soils by the application to the surface soil. In one such embodiment a liquid as described above is applied and in another a dry powder is spread on the soil. This may also be used to control mud or to solidify ground that is to soft to otherwise work. Such applications will be apparent to one of skill in the art.

The following examples are included to demonstrate preferred embodiments of the invention. It should be appreciated by those of skill in the art that the techniques disclosed in the examples which follow represent techniques discovered by the inventors to function well in the practice of the invention, and thus can be considered to constitute preferred modes for its practice. However, those of skill in the art should, in light of the present disclosure, appreciate that many changes can be made in the specific embodiments which are disclosed and still obtain a like or similar result without departing from the scope of the invention.

EXAMPLE 1

In extremely wet sand in a backfilled lagoon in a coastal region in Asia, the normal digging of drilled shaft foundations for a high rise residential development was undertaken.

The 0.8 meter diameter digging bucket was used and the excavated walls were not considered stable enough to insure an adequate time frame for the placement of reinforcing steel and concrete. The contractor first elevated the casing by 1.5 meters. Approximately 2 meters above the water table was estimated. The casing was kept full of

a polymer based fluid at all times. The hole was experiencing modest sloughing at 16 to 28 meters of depth.

At 28 meters onward, a stable marine clay was encountered. At the toe of the hole at 37 meters the friction pile was at full depth. The fluid used was at an adjusted pH of 10-12 with a SlurryPro® CDP and SlurryPro® MPA system from KB Technologies Ltd. of Chattanooga, Tennessee in use. The CDP polymer was used at 1.1 kilograms per cubic meter of fluid and the MPA was used at 0.01 kilograms per cubic meter of fluid. The Marsh Funnel Viscosity was maintained at 75 seconds/quart. The system was pronounced adequate under these severe running sand conditions with a concrete overbreak of approximately 25%. The area of this site was contaminated with a ruptured and abandoned sewer line with organic matter in the upper layers of the sandy zones.

EXAMPLE 2

At the same construction site as example 1 above, another excavation in the same area within 25 meters of the first example was drilled. Again 0.8 Meter diameter was excavated experiencing the same loose wet sand followed by marine clay and ending in a sandy clay at 37 meters down. The same fluid system as in example 1 was used as supplied by KB Technologies Ltd. of Chattanooga, Tennessee. On this pile the addition of 2 drums of sodium silicate from a 40% solution was done at just below the casing at approximately 16 meters. This put a concentration of 4.1% sodium silicate into the fluid volume, w/w.

The resulting shift in the specific gravity of the excavation fluid system was an increase to 1.05 from an initial value of 1.01. The sandy deposits from the digging bucket were noticeably better formed with a "drier" appearance. They had a firmer feel and heaped up on the spoils pile in a more manageable way. The cleaning of the hole was improved with a reduction of sediment. The concrete overbreak was 19%. The addition of the sodium silicate seemed to have a chemical effect on the sandy zone and immediately facilitated a successful excavation beyond the weighting effect on the fluid itself.

EXAMPLE 3

Another example in the same extremely sandy are was performed where both sodium silicate and sodium aluminate were added to a very runny sandy zone at 16 to 30 meters below the surface. The same fluid was in use as in examples 1 and 2 above.

The introduction of one drum of sodium silicate weighing 260 kilograms was placed at about 20 meters down into the drilling of this hole. This was allowed to mix for 5 minutes then a 25 kilogram bag of sodium aluminate was added to the top of the hole and drilling was continued after a 5 minute mix. The spoils from the bucket were noticeably drier and felt slightly warmer to the touch, An estimate of 3 degrees Centigrade rise in temperature was estimated for the material taken from the bucket. The hole was taken to full depth at 37 meters and cleaned in the usual way with about 0.4 meter of sediments. The concrete overbreak was 16%. This was considered an improvement.

EXAMPLE 4

At another site in Asia sodium silicate was used with a fluid like example 2 above. Here a 1.8 Meter pile was drilled for a railroad bridge. The pile was required to go through approximately 40 meters of totally decomposed granite and finished as a friction pile in a sandy clay at 45 meters depth.

During the drilling the t.d.g., totally decomposed granite, tends to stress relief itself. This makes for a small tightening of the hole and a need to keep small amounts of the sandy material from getting loose in the hole. The fluid level was maintained as high as practical, with a 1.5 meter elevated casing. The water table was approximately 6 meters down. The Marsh Funnel Viscosity for the excavation was held at 75 seconds/quart or higher at a pH of 12. Two drums of sodium silicate, 40%, were added at the 30 meter level. The casing for this hole was 21 meters. The specific gravity was increased 0.03 to 1.04. Of greater importance, the reactive material, sodium silicate, was delivered to the zone that most needed protection down to the 40 meter level. Beyond 40 meters a more clay bearing layer was encountered.

The resulting hole was drilled to depth and cleaned. The steel was placed and concrete overbreak was only 3%, typical for this sort of formation, The improvement to 0.7 meter of bottom sand sediment layer at the toe was an improvement. from the 1 to 2 meters experienced normally.

EXAMPLE 5

The following procedure was followed to solidify a zone near the surface, after the removal of obstructions in preparation for the placement of a guide-wall for a diaphragm wall or slurry wall. The excavated area is filled with a mixture of sand, gravel ,sea shell and sandy soil delivered to the site from a mixing system in water with a 12.0% sodium silicate content w/w added by the mix plant from a 40% grade of sodium silicate to create a premixed part one of a soil repair system. At the point of addition to the excavation, the slurry is approximately 75 to 85% solids content from the solid material. To this is co-added by a metering pump, a small stream of 6% sodium aluminate based solution from a commercially available source that has been enriched with sodium hydroxide by 4% of the formula weight of the excavation filling mixture; this is part two. The mixture is allowed to set for a period of one hour and then a solid sandstone material results suitable to allow the placement of a guide-wall.

This example is a replacement for a lean-mix concrete system that is produced from a calcium free formulation. The advantage is that when excavated the spoils will not harm the fluid integrity and the cost is justified since a damaged fluid will not result.

EXAMPLE 6

22.2g of a 0.1% pH=11+ SlurryPro CDP fluid was mixed with 181.4g of blasting sand (Foster Dixiana). To this was added 60.0g of QUIKRETE all purpose gravel and 56.0g of 40% grade sodium silicate. The material was agitated to ensure complete mixing of components. A separate solution of 51.2g of 45% grade sodium aluminate and 12.6g of 50% grade sodium hydroxide was prepared and then added to the previous mixture with vigorous agitation for 15 seconds. Immediately the mixture was poured into a 3 inch

round polystyrene cap. Before the material solidified a knife was used to level off the surface evenly with the edges of the cap. The newly formed mixture was unable to flow within 2 minutes of mixing. After one hour the mixture had solidified to a ridged beige solid. After 24 hour the solid "puck like" structure was removed from the cap. This material was subjected to a compression test and was able to support over 200 psi of applied force. Material calculated to be as 76.2% solids.

EXAMPLE 7

100.0g of blasting sand, 1.0g of finely ground SlurryPro CDP, 2.0g citric acid, 3.8g of sodium bicarbonate, and 0.65g of 2-hydroxybenzoic acid were combined and mixed until a uniformly distributed dry mixture was observed. This material was used to provide two 53.5g samples. A 50% w/w solution of 40% grade sodium silicate and Chattanooga City water was mixed. To a beaker was added 45.5g of the 50% sodium silicate/water solution. Without any agitation or mixing, one dry mixture sample was added to this beaker. The second dry mixture sample was placed in a second beaker. To this beaker was added 53.0g of the 50% sodium silicate/water solution. Both samples were completely submerged under the silicate/water solution. After 1 hour each sample was a thick sticky paste. While after 24 hours, each sample had solidified into a beige mass underneath the surface of the fluid.

EXAMPLE 8

100.0g of blasting sand, 13.0g of sodium aluminate powder, and 6.0g of sodium hydroxide small beads were mixed until a uniform mixture observed. This material was split into two 59.5g samples. One sample was added directly to 47.8g of a 50% sodium silicate and water solution (see example #7). The second sample was added to 52.2g of a 50% sodium silicate/water solution. Within 15 minutes each sample had formed a semisolid mass. After 24 hours, each sample had developed further into a hardened mass.

EXAMPLE 9

A mixture of 181.4g of blasting sand, 22.2g of 0.1% pH=11+ SlurryPro CDP fluid, and 56.0g 40% grade sodium silicate were combined and allowed to sit for 24 hours. This mixture was added to a beaker filled with 0.1% pH=11+ SlurryPro CDP fluid and settled to the bottom of the beaker. A solution of 51.2g of 45% grade sodium aluminate and 12.6g of 50% grade sodium hydroxide was mixed. The sodium aluminate and sodium hydroxide solution was then injected into the lower portions of the sand mixture through a syringe. Within 10 minutes the sand layer had increased 20+°F in temperature and a thick paste was forming around the area of injection. After 24 hours 90+% of the sand mixture was a combination of solid and gelled areas.

EXAMPLE 10

A beaker was filled with 0.1% pH=11+ SlurryPro CDP fluid. To this was added 60.0g of 40% grade sodium silicate which settled to the bottom of the beaker. A anhydrous mixture of 100g blasting sand, 10g sodium hydroxide flake, and 15g sodium aluminate powder were mixed until uniformity was observed. This anhydrous mixture was added to the beaker and settled on the bottom. After one hour the sand mixture was a gelled mass. After 24 hours the sand layer was a thick paste.

EXAMPLE 11

In a North East US city, a large cut and cover tunnel project requires that panels be installed near the original shoreline in areas of extreme fill and in an area where wood timbers from older foundations are numerous. The test panel selected is a secondary panel just north of a panel that had been difficult previously due to soft zones and wood piling obstructions. This panel was expected to be soft as well.

A photograph was taken of pretrenched area before fluid was added. Ground water was rushing in near bottom of pretrench area. Fluid out of hose supply to fill trench was adjusted to 50 sec by Marsh Funnel Viscosity test at pH 10+. The initial excavation was mostly digging wood and rocks--no clay. The fluid contained tap water

- 1 for make-up and 1.0 kilogram per ton SlurryPro ®CDP, dry vinyl resin polymer,
- 2 supplied by KB Technologies of Chattanooga, Tennessee. The pH was adjusted with
- 3 alkalis to pH 10 -12. The fluid level in the excavation guide wall was holding but the
- 4 spoils were very soupy in nature and poorly shaped. Inbound fluid at 48 seconds was
- 5 measured. Gravel, marine shells and some marine clay were in the spoils. SlurryPro
- 6 CDP was then added at the hole in dry form, 1/3 of a 44 pound bag of CDP, at the 17
- feet depth. Now, 57 seconds in hole viscosity and pH 11+.
- By observing a painted mark on the side of panel I-beam during 30 minute delay
- 9 for chisel work to remove concrete on the panel I-beam, the fluid level was steady. No
- leaks at this time. The bucket is still bring up concrete and lean mixture. A top sample at
- 40 seconds /quart was measured due to concrete contamination. More CDP approx. 1/2
- bag (22 pounds) is added.
- The digging of concrete/lean mix at 20 feet continued. Incoming slurry was 55
- seconds and pH 10. Added 1 gal of SlurryPro ®MPA, a cationic polyamide resin
- supplied by KB Technologies, via a bag bomb to help seal the walls.
- Viscosity now at 63 seconds. at 20 feet of depth. MFV in hole is 56 seconds
- and added another MPA bag bomb. Fluid level in the trench is holding steadily. At 33
- feet mostly clay with gravel and sand we added another MPA bag bomb.
- 19 Continued to dig well. The hole was left open over the weekend.
- To begin the next week, the crew began excavation, still chiseling--middle 34
- feet with a 52 sec in hole viscosity. Evidence of a unstable layer was expected at 35
- 22 to 40 down.
- Added Instafreeze® System to the panel: 3 drums--totaling 1980 pounds--
- Instafreeze additive (40% sodium silicate 3.22:1 SiO2: Na2O)-- 1 drum -660 pounds--
- Instafreeze C-2(sodium aluminate-40%) and 1 drum--660 pounds-- of Protek 100,
- 26 KOH, 45% alkalinity source) and continued to dig. At 40 feet all was in control and
- stable, the spoils were holding shape and no evidence of caving or heaving is noticed.
- Coden® --sonic logging device--Okay except for a small zig-zag at 30+ feet. The
- 29 excavation has straight sides through the difficult zone. 53 seconds in the hole viscosity
- is maintained. Finally finished chiseling shape on Coden shows only a 1-2 foot nick in

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walls at sand layer. At 88 feet going and going to 90.5 feet into in the rock socket --some chisel damage up higher due to lean mix and concrete removal.

Hole finished: Concrete placement: 330 cubic yards for a 37.9 % overbreak.

Collapse avoided---- overall Okay. W=3.82 L 19.1 D=89 ft3=6493.62 48,572.26 gallons == 239.27 yds3 The hole was done in three days of active operation and stayed open over a weekend holiday.

In view of the above disclosure, one of ordinary skill in the art should understand and appreciate that one illustrative embodiment of the present invention includes an excavation fluid composition useful for enlarging a cavity in the earth. Such a fluid includes a mixture of a synthetic polymer and sodium silicate, preferably in water, organic fluids or mixtures thereof. The fluid is formulated so as to enable the fluid in contact with unstable or sandy soils in the selected areas of the excavation to react and form silicate-based derivatives with lesser solubility, and movement. Thus the fluid improves soil stability at the excavation wall and permits the excavation process to continue. One preferred embodiment of the present illustrative embodiment the fluid further includes an alkalinity source, said alkalinity source being present from 0.01% to 10.0% by weight of the excavation fluid. Alternatively, the fluid may be formulated so as to have a pH value of between 4 and 13. In another preferred illustrative embodiment, the synthetic polymer is formulated from one or more monomers selected from: a. acrylamide, methacrylamide, acrylic acid, methacrylic acid, maleic acid, fumaric acid; b. maleic anhydride, methacrytic anhydride, itaconic acid, acrylic acid dimer(BCEA), Misopropenylbenzyl dimethyl isocyanate and the nonionic associative monomer derivatives, esters or urethane, so produced containing nonionic surfactant starting materials prepared from ethylene oxide and/or, propylene oxide and/or, butylene oxide and/or C_1 to C_{20} alkyl alcohols and/or C_8 to C_{12} alkyl phenols; c. itaconic acid, vinylsulfonic acid, styrene sulfonic acid, 2-acrylamido-2- methylpropane sulfonic acid, methallylsulfonic acid, vinyl acetic acid, 4- methylpentenoic acid, allylacetic acid, B-hydroxyethylacrylate, x-haloacrylic acid; d. M- isopropenylbenzyl dimethyl isocyanate and its nonionic derivatives prepared from alkyl alcohols; e. methylenebisacrylamide, N-

methylol acrylamide, triallyl cyanurate, vinyl crotonate, divinylbenzene, allyl methacrylate; f acrylic acid esters of sucrose, hexallyl sucrose, trimethylolpropane triacrylate, ethylene glycol diacrylate, diethylene glycol diacrylate, ethylene glycol dimethacrylate, and the like; g. methacrylic anhydride esters or maleic anhydride esters of sucrose, sorbitol, sorbitol esters with fatty acids; h. guar gum, starch, ethylated starch, oxidized starch, starch fatty acid esters, dodecylsuccinic anhydride modified starch, agar gum, xanthan gum, arabic gum or galacto-mannin derivatives prepared from methacrylic anhydride or maleic anhydride or M-isopropenylbenzyl dimethyl isocyanate resulting in hybrid monomers; i. vinyl acetate, N-vinyl formamide, N-vinyl acetamide, N-vinyl pyrolidone, styrene, butadiene, isoprene, chloro-butadiene, vinyl chloride, vinylidene chloride, C₁ to C₂₀ acrylate and methacrylate esters; j. methacryloxyethyl dimethylamine, methacrylamido propyl dimethylamine, dimethyl diallyl ammonium chloride, diethyl diallyl ammonium chloride, and their methyl sulfate and methyl chloride derivatives and water soluble or dispersible salts and combinations thereof.

Another illustrative embodiment of the present invention includes a method of stabilizing the wall of an earthen excavation including: placing in the earthen excavation a digging fluid, composed of a polymer and sodium silicate. Preferably the composition is formulated so as to enable the fluid in contact with unstable or sandy soils in the selected areas of the excavation to react and form silicate-based derivatives with lesser solubility, and movement and thus improve soil stability at the excavation wall.

In one preferred illustrative embodiment, the digging fluid further includes an alkalinity source in an amount from 0.01% to 10.0% by weight of the excavation fluid.

It is further preferred that the polymer be composed of one or more monomers selected from: a. acrylamide, methacrylamide, acrylic acid, methacrylic acid, maleic acid, fumaric acid; b. maleic anhydride, methacrylic anhydride, itaconic acid, acrylic acid dimer(BCEA), M-isopropenylbenzyl dimethyl isocyanate and the nonionic associative monomer derivatives, esters or urethane, so produced containing nonionic surfactant starting materials prepared from ethylene oxide and/or, propylene oxide and/or, butylene oxide and/or C₁ to C₂₀ alkyl alcohols and/or C₈ to C₁₂ alkyl phenols; c. itaconic acid, vinylsulfonic acid, styrene sulfonic acid, 2-acrylamido-2- methylpropane sulfonic acid,

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methallylsulfonic acid, vinyl acetic acid, 4- methylpentenoic acid, allylacelic acid, Bhydroxyethylacrylate, x-haloacrylic acid; d. M- isopropenylbenzyl dimethyl isocyanate and its nonionic derivatives prepared from alkyl alcohols; e. methylenebisacrylamide, Nmethylol acrylamide, triallyl cyanurate, vinyl crotonate, divinylbenzene, allyl methacrylate; f. acrylic acid esters of sucrose, hexallyl sucrose, trimethytolpropane triacrylate, ethylene glycol diacrylate, diethylene glycol diacrylate, ethylene glycol dimethacrylate, and the like; g. methacrylic anhydride esters or maleic anhydride esters of sucrose, sorbitol, sorbitol esters with fatty acids; h. guar gum, starch, ethylated starch, oxidized starch, starch fatty acid esters, dodecylsuccinic anhydride modified starch, agar gum, xanthan gum, arabic gum or galacto-mannin derivatives prepared from methacrylic anhydride or maleic anhydride or M-isopropenylbenzyl dimethyl isocyanate resulting in hybrid monomers; i. vinyl acetate, N-vinyl formamide, N-vinyl acetamide, N-vinyl pyrolidone, styrene, butadiene, isoprene, chloro-butadiene, vinyl chloride, vinylidene chloride, C_1 to C_{20} acrylate and methacrylate esters; j. methacryloxyethyl dimethylamine, methacrylamido propyl dimethylamine, dimethyl diallyl ammonium chloride, diethyl diallyl ammonium chloride, and their methyl sulfate and methyl chloride derivatives and water soluble or dispersible salts and combinations thereof. Optionally the digging fluid is composed of: a. a synthetic polymer, b. sodium silicate being 0.1% to 50.0% of the fluid composition and c. sodium hydroxide being 0.01% to 10.0% of the fluid composition

Yet another illustrative embodiment of the present invention includes a process of improving boreholes, trenches or other excavations' dimensional stability by including sodium, potassium or other soluble silicate into a mixture of water, soils, sands and a synthetic polymer water based fluid during excavation and enlargement. Such an illustrative process is preferably be carried out when the drilling fluid is a synthetic polymer fluid between the pH of 4 and 13. In one illustrative embodiment, the mixed fluid in the excavation, in a range of density of 1.01, as with fresh polymer fluid prior to contacting the excavation, up to 1.20 g/cc after the silicate added fluid has reacted with the slurry system in the active excavation cavity. Preferably the silicates are at a mole ratio of SiO₂ to M₂O of 1:1 to 4:1, respectively wherein M is an alkali metal. More

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preferably, the polymer fluid is a synthetic polymer or polymers based fluid containing at least one polymer prepared from the list of monomers: acrylamide, methacrylamide, acrylic acid, methacrylic acid, maleic acid, fumaric acid; maleic anhydride, methacrylic anhydride, itaconic acid, acrylic acid dimer(BCEA), M-isopropenylbenzyl dimethyl isocyanate and the nonionic associative monomer derivatives, esters or urethane, so produced containing nonionic surfactant starting materials prepared from ethylene oxide and/or, propylene oxide and/or, butylene oxide and/or C1 to C20 alkyl alcohols and/or C8 to C₁₂ alkyl phenols; itaconic acid, vinylsulfonic acid, styrene sulfonic acid, 2acrylamido-2-methylpropane sulfonic acid, methallylsulfonic acid, vinyl acetic acid, 4methylpentenoic acid, allylacetic acid, B-hydroxyethylacrylate, x-haloacrylic acid; Misopropenylbenzyl dimethyl isocyanate and its nonionic derivatives prepared from alkyl alcohols; methylenebisacrylamide, N-methylol acrylamide, triallyl cyanurate, vinyl crotonate, divinylbenzene, allyl methacrylate; acrylic acid esters of sucrose, hexallyl sucrose, trimethylolpropane triacrylate, ethylene glycol diacrylate, diethylene glycol diacrylate, ethylene glycol dimethacrylate, and the like; methacrylic anhydride esters or maleic anhydride esters of sucrose, sorbitol, sorbitol esters with fatty acids; guar gum, starch, ethylated starch, oxidized starch, starch fatty acid esters, dodecylsuccinic anhydride modified starch, agar gum, xanthan gum, arabic gum or galacto-mannin derivatives prepared from methacrylic anhydride or maleic anhydride or Misopropenylbenzyl dimethyl isocyanate resulting in hybrid monomers; vinyl acetate, Nvinyl formamide, N-vinyl acetamide,, N-vinyl pyrolidone, styrene, butadiene, isoprene, chloro-butadiene, vinyl chloride, vinylidene chloride, C₁ to C₂₀ acrylate and methacrylate esters; methacryloxyethyl dimethylamine, methacrylamido propyl dimethylamine, dimethyl diallyl ammonium chloride, diethyl diallyl ammonium chloride, and their methyl sulfate and methyl chloride derivatives and water soluble or dispersible salts and combinations thereof.

In carrying out the illustrative method, it is preferred that the reaction of the silicate salts with the synthetic polymer fluid, soils, sands and other materials in the excavation cavity to form tackified masses which assist in the creation of a superior fluid loss barrier at the formation interface. It is further preferred that the included silicate salts

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assist in the dispersion and carrying of colloids within the polymer slurry thus assisting in increasing the specific gravity of the slurry increased solids dispersion throughout the active synthetic polymer slurry. One of skill in the art will also note that the creation of semi-solid tackified masses and the improvement in the creation of a pressure transfer barrier between and slightly within the formation and the slurry resulting in improved fluid loss control combined with the increased differential pressure within the slurry causing superior excavation side wall support a more gauge and superior performing foundation element is created.

It should also be appreciated by one or ordinary skill in the art that a present illustrative embodiment of the present invention includes an anhydrous acid solidification mixture. One illustrative anhydrous acid solidification mixture preferably includes: a structural material, a polymer component; an accelerator compound, an acidic component, and a silicate component. Preferably the structural material being used provides stability, strength, support, foundation, or volume to the solidification mixture and being selected from: sands, soils, clays, pebbles, cobbles, marble, granite, stones, gravel, rocks, bentonite, cement, polymer fibers, sandstone and combinations thereof. The accelerator compound is preferably selected from chemicals capable of producing carbon dioxide in acidic environments, chemicals capable of producing chlorine gas in acidic conditions, inorganic chloride salts, inorganic sulfate and inorganic sulfite salts. More preferably, the accelerator compound is selected from the group consisting of potassium and sodium salts of hydrogen carbonate, potassium and sodium salts of carbonate, sodium and potassium hypochlorite, and combinations thereof, and wherein said acidic components is selected from the group consisting of citric acid, the salts of citric acid, sulamic acid, and combinations thereof. The acidic component is preferably selected from solid chemicals between the pH of 4 and 13. The silicate component is preferably selected from sodium orthosilicate, sesquisilicate, metasilicate, disilicate and combinations thereof.

While the apparatus, compositions and methods of this invention have been described in terms of preferred or illustrative embodiments, it will be apparent to those of skill in the art that variations may be applied to the process described herein without

- departing from the concept and scope of the invention. All such similar substitutes and
- 2 modifications apparent to those skilled in the art are deemed to be within the scope and
- 3 concept of the invention as it is set out in the following claims.